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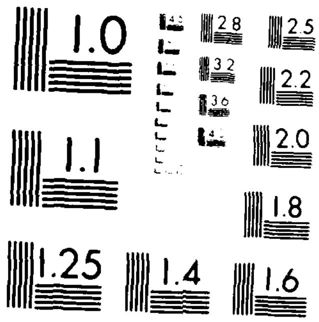

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SHELF LIFE DEGRADATION OF SELECTED ANAEROBIC THREADLOCKING ADHESIVES AT VARIOUS STORAGE TEMPERATURES

PAUL R. BERGQUIST and STANLEY E. WENTWORTH
POLYMER RESEARCH DIVISION

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ABSTRACT

This study addresses the shelf life degradation of three commercially available anaerobic adhesives maintained at 0°C, 22°C, and 50°C. A titrimetric procedure was developed to monitor the change in the adhesive peroxide initiator concentration over an 18 month period. The change was then related to a corresponding decrease in the breakaway and prevailing torque strengths of bonded nuts and bolts prepared with the aged adhesives. It was determined that elevated temperature storage caused degradation of the adhesives and that the titrimetric method could be useful as a quality control method where such storage may be unavoidable.

CONTENTS

INTRODUCTION

Mechanical fasteners often loosen as a result of prolonged vibration and intermittent shock. Overcoming this problem through the use of anaerobic adhesives represents a 50 percent cost savings over the mechanical split lock washer and additional savings on the maintenance time required to retighten loose bolts or fasteners.

This study deals with the shelf life degradation of anaerobic adhesives prior to application. It is an extension of an earlier project that addressed the chemical composition of some 45 commercial anaerobic adhesive and sealant formulations.[*] This earlier work indicated that the shelf life degradation of the uncured adhesives was very likely to influence the curing behavior of the adhesive and the mechanical properties of adhesively bonded specimens.

Anaerobic adhesives are complex formulations that contain an unsaturated oligomeric base resin and a peroxidic initiator. In addition, they contain fillers, reactive diluents, accelerators, inhibitors, bond strength enhancers, and viscosity modifiers. Subtle changes in the concentration, the number of components present, or the nature of the components, can result in profound changes in the ability to cure, as well as the physical properties of the cured adhesive. Both qualitative and quantitative analyses of each component would be required to thoroughly study just one adhesive formulation. Even then, it would be difficult, if not impossible, to determine which component or combination of components directly affected the mechanical strength of the cured adhesive.

However, it was felt that monitoring the concentration of the peroxide cure initiator, the least stable and most important component, would provide a good indication of deterioration of the adhesive, especially when correlated with breakaway and prevailing torque data. No data was available as to the rate or effect of such degradation on subsequent bonding performance. Thus, a detailed study was undertaken to determine the effect of shelf-storage on chemical composition and mechanical properties of several representative anaerobic adhesive formulations.

After evaluating various standard methods[1-5] and conducting a literature survey on methods for the analysis of organic peroxides, it was concluded that, without modification, none of them would meet our requirements for a quick, simple, and accurate means of determining organic peroxides in complex mixtures. Therefore, a modified wet-potentiometric titration method was developed.[5]

*WENTWORTH, S.F. Unpublished work.

1. *Assay of Organic Peroxides, Method A.* 1979 Annual Book of ASTM Standards, ASTM E-298, part 30, p. 830-831
2. *Assay of Organic Peroxides, Method B.* 1979 Annual Book of ASTM Standards, ASTM E-298, part 30, p. 832-833.
3. *Standard Test Method for Traces of Peroxides in Organic Solvents With N,N'-Diphenyl-p-Phenylenediamine.* 1979 Annual Book of ASTM Standards, ASTM E-440, part 30, p. 956-959.
4. *Standard Test Method for Trace Amounts of Peroxides in Organic Solvents.* 1979 Annual Book of ASTM Standards, ASTM E-299, part 30, p. 834-837.
5. *Assay of Di-Ter-Butyl Peroxide by Gas Chromatographic Analysis.* 1979 Annual Book of ASTM Standards, ASTM E-475, part 30, p. 984-987.
6. CHIN, W. and BERGQUIST, P.R. *Method Development for the Analysis of Cumene Hydroperoxide in an Anaerobic-Adhesive System.* To be submitted for publication as a technical report.

EXPERIMENTAL

Sample Acquisition and Storage

Six 50 ml plastic bottles of each of the three commercial adhesives presented in Table 1 were purchased from local distributors. The adhesives to be studied were selected as representative of commercial threadlocking formulations. Two of the adhesive formulations (Adhesives B and C) contain cumene hydroperoxide as the peroxide initiator, while all three anaerobic adhesives have tetraethylene glycol dimethacrylate present as the base resin.

Table 1. ADHESIVES STUDIED

Adhesive	Viscosity	Color*	Type*	Grade*
A	150 cps	Blue	I	J
B	900-1300 cps	Blue	II	N
C	900-1300 cps	Blue	II	N

*The classification of type and grade are based on MIL-S-46163A.

After initial determination of the peroxide concentration and torque values, the adhesives were stored in controlled environments at possible depot storage temperatures. Three bottles of each adhesive were stored at 50°C, two bottles of each at 22°C, and one bottle of each at 0°C. Samples were withdrawn and analyzed at intervals (frequency and duration dependent on initial results) to determine how the composition was changing. Mechanical test specimens were prepared and tested concurrently.

Torque Measurements

The torque measurements consisted of bonding a nut and bolt and testing the torque required to break the bond and maintain rotation after break. The mechanical test specimens were prepared and tested according to MIL-S-46163A, sections 4.6.2.1.1 and 4.6.2.1.2. The resulting breakaway torque and prevailing torque based on five specimens were measured at an initial quarter turn and as the average of the next four quarter turns. The values are reported in inch-lbs.

Peroxide Determination

The bottles were well shaken before sampling. Approximately 0.50 grams of sample was weighed into a 250 ml erlenmeyer flask before adding 25 ml of isopropanol. The flask was capped and swirled. Four milliliters of glacial acetic acid was added, and the flask was again capped and swirled before adding 10 ml of saturated sodium iodide in isopropanol. The flask was connected to a condenser and the contents allowed to reflux with stirring for 7 minutes. The condenser was washed down with 5 ml of water. Finally, the flask was cooled in cold water to room temperature. The total time from start to finish was 10 minutes.

The solution was titrated with a 0.01 N standardized sodium thiosulfate solution using a redox electrode-pH meter setup. A blank of the above reagents was run. The calculation of percent peroxide is as follows:

$$\text{\% Peroxide Content (Calculated as Cumene Hydroperoxide}^{*}) = \frac{(A-B)(N)(M.W.)(100)}{(WGT)(2C)(1000)}$$

where

A = milliliters of $Na_2S_2O_3$ titrant required for sample
B = milliliters of $Na_2S_2O_3$ titrant required for blank
C = number of peroxide groups in the peroxide compound
N = normality of $Na_2S_2O_3$
M.W. = molecular weight of the peroxide compound
WGT = gram weight of sample used

The accuracy and precision of the titration method were established by analyzing a model anaerobic formulation consisting of tetraethylene glycol dimethacrylate and a known concentration of cumene hydroperoxide. Based on the average deviation for this method, the percentage of error for the experimentally determined value is $\pm 1.3\%$

Instrumentation

Corning Model 150 pH Meter/ion meter, automatic

Orion electrode, combination, redox (977800)

APCO-Mossberg Torque Wrench RDI-150

APCO-Mossberg Torque Wrench RDI-75

RESULTS AND DISCUSSION

After the initial baseline data was determined, the adhesives were placed in storage in controlled environments simulating extreme service storage conditions. The initial characterization and mechanical test data is presented in Table 2.

Table 2. ADHESIVE BASELINE DATA

Adhesive	Peroxide Concentration (%)	Breakaway Torque (inch-lbs)	Prevailing Torque (inch-lbs)
A	1.47	118	38.4
B	1.59	115	39.6
C	2.59	107	36.6

*Cumene hydroperoxide has a molecular weight of 152 with one peroxide group in the molecule.

$50^{o}C$ Storage

The anaerobic adhesives maintained at $50^{o}C$ are the only adhesives to show any real change in percent reported cumene hydroperoxide concentration and torque values during this 574 day study. Adhesive A (Figures 1 and 2) was the first material to exhibit a change in both peroxide concentration and torque values. The initiator concentration decreased from an initial average value of 1.47% to 0.82% after 127 days. Illustrated in Figure 2 is the decline in the breakaway torque for materials stored at $50^{o}C$. As can be seen, the breakaway torque for Adhesive A drops from an initial average value of 118 to 14 inch-lbs after 127 days. The prevailing torque (Figure 3) declined from 38.4 to 4 inch-lbs in the same time period.

Adhesive C also exhibited a significant change in torque values (Figures 2 and 3). The peroxide concentration steadily decreased from an initial value of 2.59% to 0.41% after 513 days. The initial breakaway torque and prevailing torque were 107 and 36.6 inch-lbs respectively. The breakaway torque remained relatively constant for initiator concentrations above 0.74%.

Adhesive B demonstrated a steady decline in peroxide concentration from an initial value of 1.59% to 0.35% after 513 days, but exhibited no discernable change in torque values. The average initial breakaway torque for Adhesive B is 115 inch-lbs with a prevailing torque of 39.6 inch-lbs.

During the course of this study, several observations were made on the adhesives in storage at $50^{o}C$. After 33 days, the bottles became oily to the touch. Some component or components appear to be migrating through the polyethylene walls of the bottle. The labeling information on the bottles of Adhesive A rubs off when touched. After 395 days, it was observed that Adhesive C had changed color from blue to pale green, an important factor, since color is used to designate strength range for application uses.

$22^{o}C$ Storage

Even though some of the anaerobic adhesives stored at $22^{o}C$ exhibit a change in peroxide concentration (Figure 4), their torque values failed to show any significant change (Figures 5 and 6). During 513 days at $22^{o}C$, the peroxide concentration for Adhesive A steadily decreased from 1.47% to 1.08%. Adhesive C displayed a very slight change in peroxide concentration from 2.59% to 2.53%.

$0^{o}C$ Storage

Except for Adhesive A, the anaerobic adhesives stored at $0^{o}C$ show no change in cumene hydroperoxide concentration or torque values. The reported cumene hydroperoxide concentration for Adhesive A had changed only slightly from 1.47% to 1.37% after 513 days.

Failure Criteria

Anaerobic adhesives are accepted by the government based upon their ability to meet the requirements set forth in MIL-S-46163A, and discarded when they have exceeded their expiration date. According to the MIL-SPEC, anaerobic adhesive products can be rejected if they deviate from the criteria in color, viscosity, flash point, smoothness and homogeneity, packaging, solubility, wettability, corrosivity, toxicity, lubricity, ultraviolet illumination, storage stability, or speed of cure.

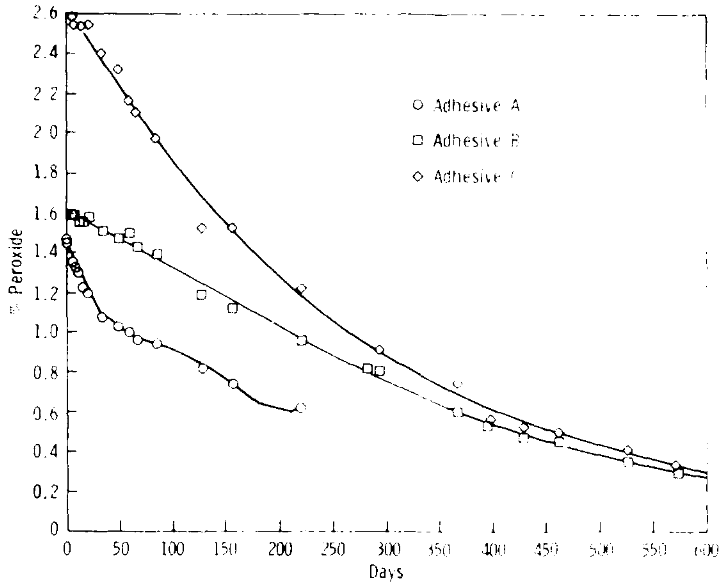

Figure 1. Change in peroxide concentration during storage at 50°C.

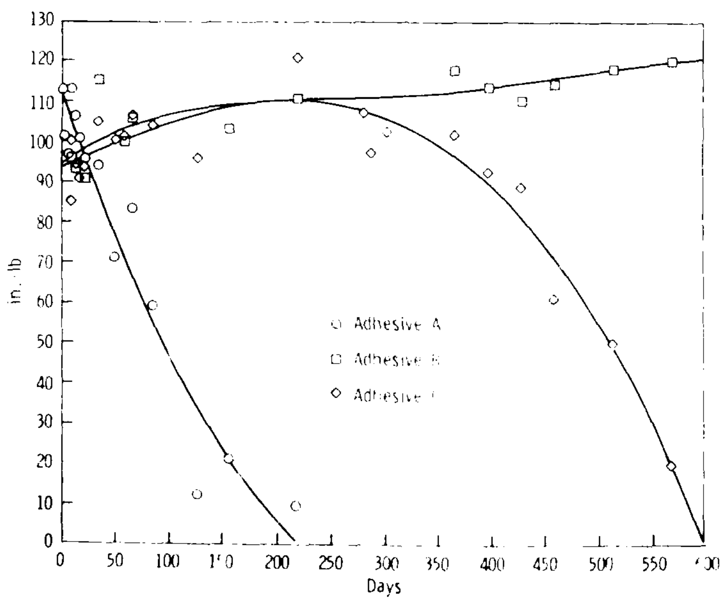

Figure 2. Breakaway torque values for anaerobic adhesives stored at 50°C.

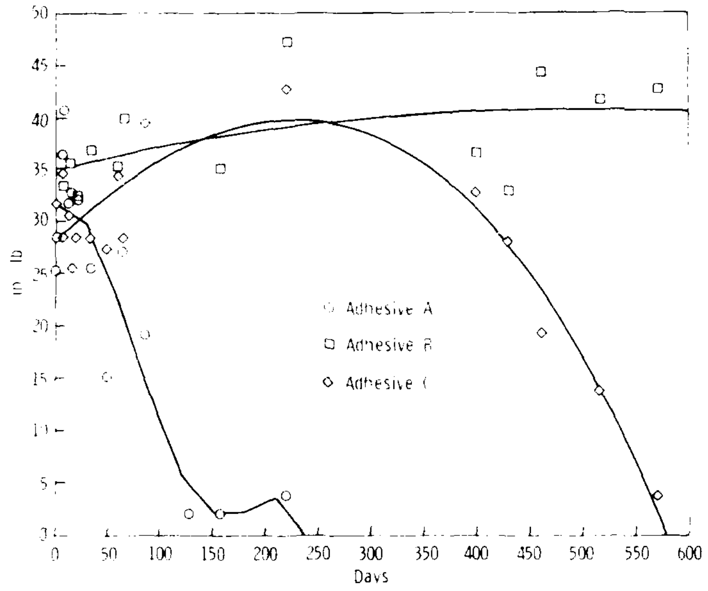

Figure 3. Prevailing torque values for anaerobic adhesives stored at 50°C.

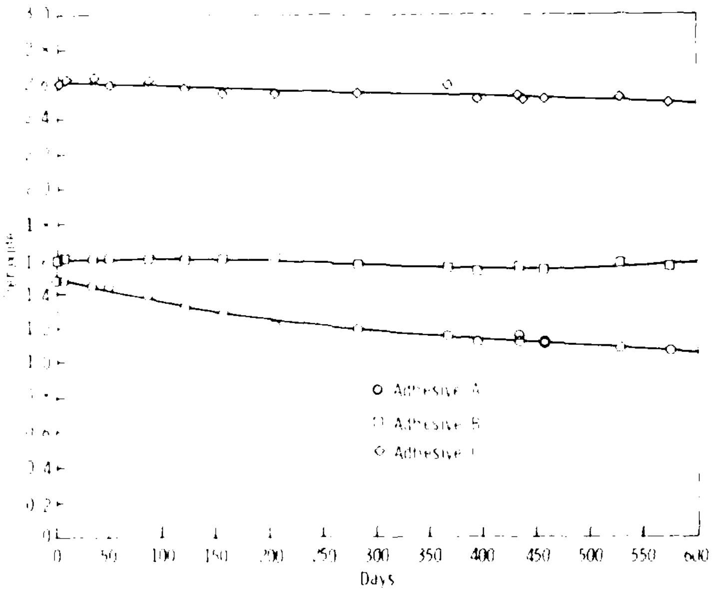

Figure 4. Change in peroxide concentration during storage at 22°C.

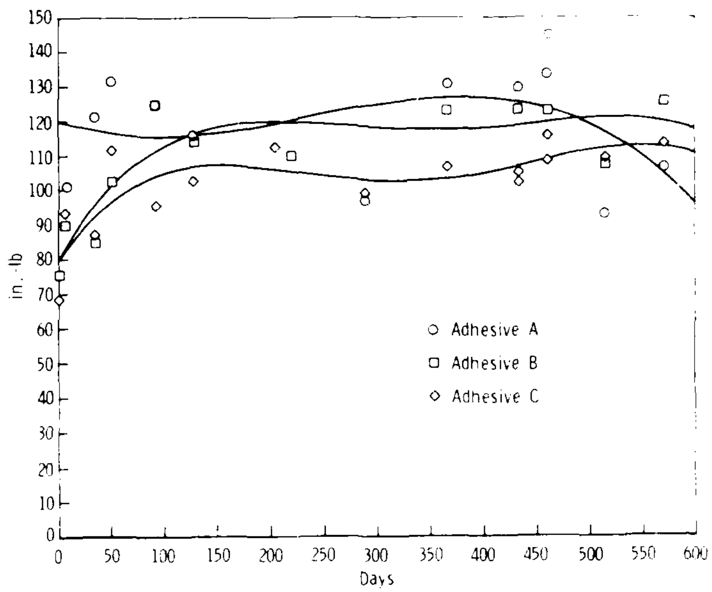

Figure 5. Breakaway torque values for anaerobic adhesives stored at 22^{o}C.

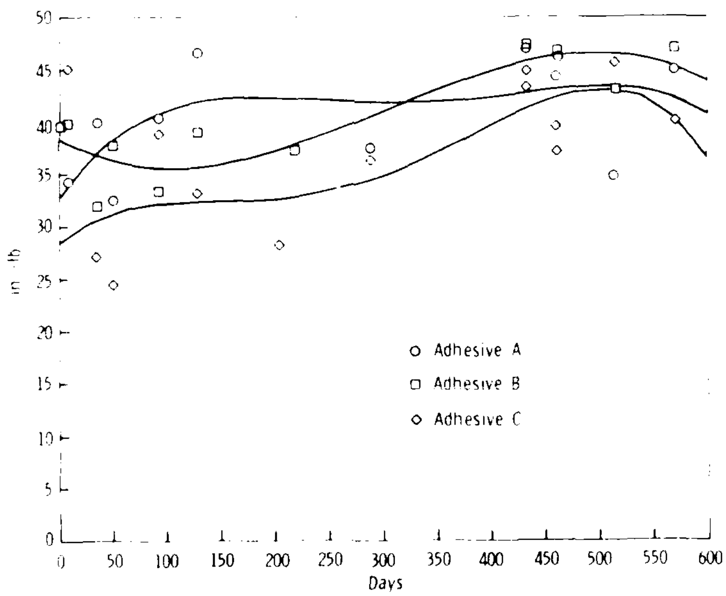

Figure 6. Prevailing torque values for anaerobic adhesives stored at 22^{o}C.

The polymerized adhesive must also meet standards for locking torque after cure, locking torque after immersion in solvents, hot strength, heat aging, low temperature torque, fluid tightness, and fungus resistance.

The point of failure or rejection of the adhesive occurs when the breakaway and prevailing torque measurements fall outside allowable ranges. The range of acceptable torque values is listed in MIL-S-46163A for adhesives classified as a particular type and grade before color coding. The type and grade are based on viscosity and locking torque. Ambiguities arise when classifying the adhesives based on viscosity, because the MIL SPEC does not specify a standard spindle size and speed. The reported viscosity, as measured by a Brookfield viscometer, varies with rotation speed and spindle size. For the adhesives used in this study, there is no clear-cut classification of these materials. Thus, the point of failure was arbitrarily taken as the point where the breakaway torque strength had dropped to 50 percent of the baseline torque.

Based on the above criteria, the failure behavior was evaluated for the adhesives stored at 50°C. Adhesive A is considered to have failed after 85 days. This corresponds to a breakaway torque of 59.2 inch-lbs, a prevailing torque of 19.2 inch-lbs, and an initiator concentration value of 0.94%. The torque values for Adhesive C remained relatively constant before showing a downward trend after 375 days. The adhesive finally reached the point of failure after 513 days at a breakaway torque of 49.6 inch-lbs with a prevailing torque of 13.9 inch-lbs and a corresponding reported cumene hydroperoxide concentration of 0.41%. The initiator concentration for Adhesive C dropped off more gradually than Adhesive A. The only adhesive to exhibit a color change was Adhesive C with a noticed change from blue to green after 395 days. It should be noted that the color change in itself is cause for rejection. Adhesive B has perhaps the most forgiving formulation. The torque values remained unchanged even though the initiator concentration reached a value of 0.35% after 572 days. The breakaway torque values appear to remain constant, and exhibit change only after the initiator concentration has dropped below its threshold value where a decrease in the mechanical properties is observed.

The peroxide concentration for the adhesives stored at 22°C changed at a very slow rate. The only adhesive at 22°C that might exhibit a change in its torque values within a reasonable period of time is Adhesive A, but this would be expected to require 4 or 5 additional months based on the apparent rate of decline (Figure 4).

The torque values (Figures 2, 3, 5 and 6) initially appear to increase. This may be due to inadequate surface preparation. The torque measurements on the nut and bolt specimens used in this study yielded values that have an average deviation as high as ± 13 inch-lbs. This resulted in the scattering of the data points and difficulty in accurately determining the exact point of failure.

SUMMARY AND CONCLUSION

The adhesive bond exhibits mechanical failure once a limiting or critical initiator concentration particular to that adhesive is reached (Figures 7-9). Only those anaerobic adhesives stored at 50°C exhibited a sufficiently rapid rate of initiator deterioration to warrant concern for shelf life. Under depot storage conditions, elevated temperatures may be unavoidable. Adhesive A would have initially passed the MIL-SPEC requirements but would have failed after 85 days of

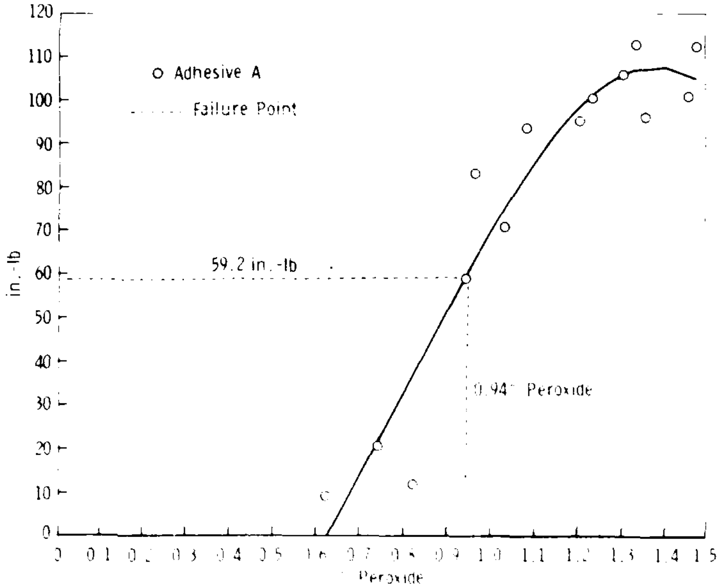

Figure 7. Breakaway torque versus peroxide concentration at 50°C.

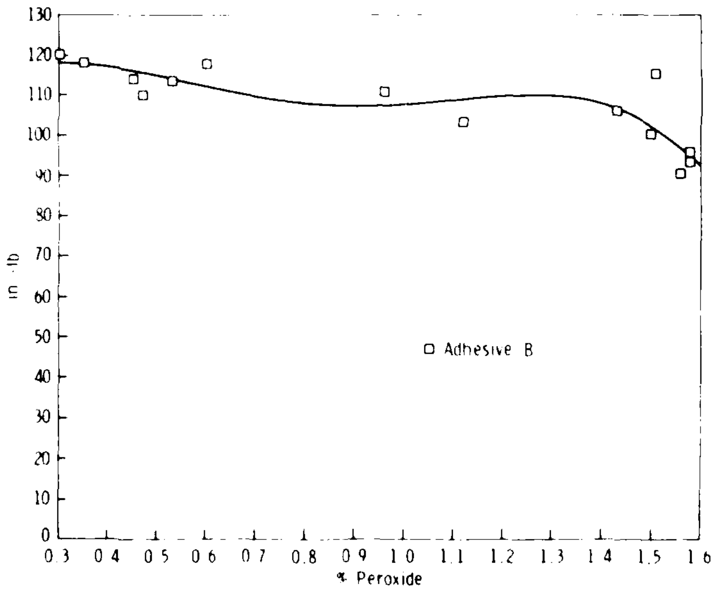

Figure 8. Breakaway torque versus peroxide concentration at 50°C.

storage* at 50°C, while Adhesives B and C had long shelf lives. The wet-potentiometric method could be useful as a quality control technique for assessing the residual shelf life of the anaerobic adhesive. The advantages of the method lie in its simplicity and speed. Although the wet-potentiometric method would not replace existing quality control tests, it would yield information relating fundamental chemical changes to observed mechanical responses. Thus, knowledge of the residual shelf life would prevent premature disposal of these relatively expensive materials. For the method of analysis to be applicable, a mastercurve depicting the relationship between the physical responses and the chemical changes must first be established. A frequently utilized adhesive with a high volume use could make the wet-potentiometric method a cost effective quality control technique.

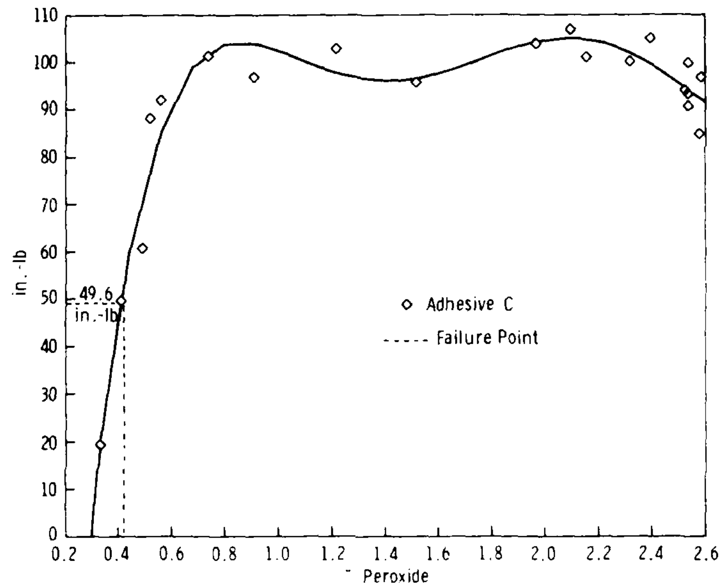

Figure 9. Breakaway torque versus peroxide concentration at 50°C.

*Since the adhesives were obtained from distributors, not from manufacturers, there is no guarantee of the exact storage conditions or time since manufacture.

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Key Words

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Adhesives
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Adhesive bonding

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Paul R. Bergquist and Stanley E. Wentworth

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Key Words

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Adhesives
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Adhesive bonding

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